Advances and Applications in Mechanical Engineering and Technology
Volume 1, Number 2, 2010, Pages 167-181

X-RAY DETECTION BY Si(Li) SEMICONDUCTOR COUNTER FOR MATERIALS INVESTIGATION

M. ROGANTE, L. PALLOTTINI and E. PETRICCI

Rogante Engineering Office
Contrada San Michele, n. 61
62012 Civitanova Marche (MC)
Italy
e-mail: main@roganteengineering.it

Via XX Settembre
n. 124, 63030 Carassai (AP)
Italy

TNX srl
Via Sommarive, n. 118
38100 Povo (TN)
Italy

Abstract

This paper concerns the Si(Li) crystal semiconductor technique adopted in the X-ray detection with main reference on material's investigation. The theoretical bases and an experimental result are reported. In particular, the analogue-digital conversion by iterative approximations method is explained, and the operation of an amplitude discriminator realized through a Schmitt trigger, the anti-coincidence circuit, and the decade counter are described, focusing on their peculiarities. The considered technique, compared with the traditional ionization chamber, requires less energy, but the cooling procedures normally employed are still presenting some difficulties. The adoption of digital electronic supports will allow future developments in the investigation of materials of industrial interest.

Keywords and phrases: X-ray, Si(Li) semiconductors, materials characterisation, analogue-digital conversion, decade counter, detector, Schmitt trigger.

Received May 4, 2010

1. Introduction

X-ray diffractometry methods are normally used for material's characterisation including crystalline phase's structures, dimensions, and residual stresses. The adoption of the X-ray for such investigations is due to their wavelength, which is comparable with the reflected ray's paths differences. In a NaCl crystal, e.g., the distance between two contiguous atoms is 2.81Å, and the wavelength normally used is 1.45Å. Si, among the semiconductor counters constitutive materials, is the most suitable for X-rays detection, while Ge represent a good detector for the γ- rays, as it is heavy enough and, therefore a good absorber. When a little quantity of Li is added - Si(Li) and Ge(Li) -, the denominations are, respectively, "silly" and "jelly". The pure Si is an intrinsic semiconductor having an elevated electrical resistivity, as a few electrons are able to overcome the gap necessary to migrate from the valence to the conduction band. The incident X-radiation, therefore, can cause the excitation and create free electrons in the conduction band and free holes in the valence band. The absorption of an X-quantum can even produce a thousand of electron-hole pairs. Electrons and holes can migrate on the opposite sides of the Si crystal, if a high difference of potentials is maintained among the same sides, thus, producing a little impulse on an external electric circuit.

The Si would behave as intrinsic semiconductor; it cannot be of *n* type, i.e., containing free electrons from a donor atom, neither of *p* type, i.e., containing free holes from an acceptor atom. In both *n* and *p* types, in fact, the free charges carriers, in their normal concentration, could overcome the few charges carriers produced by the X-rays.

The original crystal is constituted by a cylinder having the size of some millimetres and, in any case, it is of *p* type, i.e., slightly doped with the boron. The Li is applied on a side and it is diffused within the crystal at high temperature, producing a Li concentration gradient through the thickness [3]. Li exists as Li ion and the free electrons, on the other side, convert the crystal into the *n* type, while the Li concentration remains high and the other side of the crystal remains of *p* type.

A high temperature difference of potentials, then is pointed on the opposite sides with the positive pole on the *n* side and the negative one on the *p* side (inverse polarization). This effect causes a moving (drift) of the Li^+ ions through the *p* side and a wide central region having a constant concentration of Li is produced. The said region is intrinsic, as it possesses equal Li and B concentrations. The crystal results virtually intrinsic, with the *p* and *n* portions confined on the surface layers [9, 10]. A field effect transistor (FET) amplifies only the little mV impulses coming from the counter. These impulses contain only the charges freed by the X-rays absorption. A disadvantage of the Si(Li) counter is that, it operates at the liquid N temperature (77°K = –196°C) in order to minimize the constant current due either to the electrons thermal excitation in the intrinsic region or to the Li thermal diffusion.

Figure 1 represents the Si(Li) counter scheme. The FET noise increases with the temperature, thus the resolution decreases. Both counter and FET, therefore, need to be cooled in liquid N. In other types of detector such as the ionization chambers, where the X-rays ionize a gas, the energy necessary to form an electron-hole pair is superior, at least, of an order of magnitude in comparison with the semiconductor case [5]. In the 90°K Si, in fact, the medium energy to form an electron-hole pair is $\sim 3.81\text{eV}$. The number of the formed pairs, at the end of the process, is directly proportional to the incidental X-ray photon energy [3, 12].

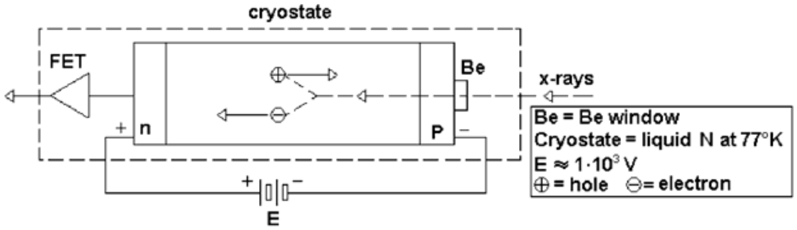


Figure 1. Scheme of the “silly” counter.

2. Theoretical

2.1. Impulses amplitude discrimination, differential discriminators and analogue-digital conversion

The electric impulse amplitude is directly proportional to the energy of the X-rays that have produced the same impulse. The impulses related to a monochromatic radiation, i.e., those having a specific energy-never have the same value, differing from the most probable medium value. A probabilistic distribution curve, thus, is obtained-approximately of the Gauss type - and the full width at half maximum of this curve is considered to evaluate the detector resolution power.

The Si(Li) detectors have a good resolution power, allowing selecting radiations with small energy differences. The purpose of the discriminator is to select the impulses having amplitude included within precise limits fixed by the user. The same discriminator receives the impulses from the FET amplifier, and it allows the passage only to those having amplitude included within fixed *max* and *min* that characterize the channel or the window width. This discriminator, therefore, gives an analogical signal directly proportional to the intensity of the incident X-rays. It is necessary, moreover, to pre-arrange the impulses number to be accumulated or the count time.

The use of Si(Li) detectors will probably increase, when cooling devices will be available, with improved or less sophisticated performances than those such as the Peltier effect based [3, 5, 7].

The differential discriminators give an output impulse only, if the input impulse amplitude is included in a precise range, as represented in Figure 2.

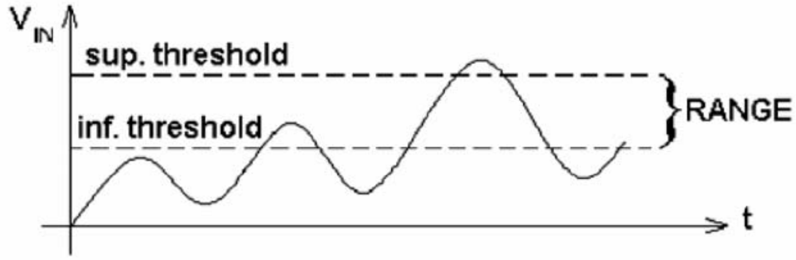


Figure 2. Input amplitude range.

After the conversion of the analogical signal into a digital one through a regulating analogue-digital converter (ADC), two integral discriminators are used, one for the inferior threshold and the other for the superior one, whose outputs are sent to an *anti-coincidence* circuit, as represented in Figure 3. In particular, the inferior and the superior thresholds in the said figure represent two digital levels and not two converters (not more analogical): They are logic and functional blocks, without which has not had sense employ the *anti-coincidence* circuit that implicates the input of two digital signals.

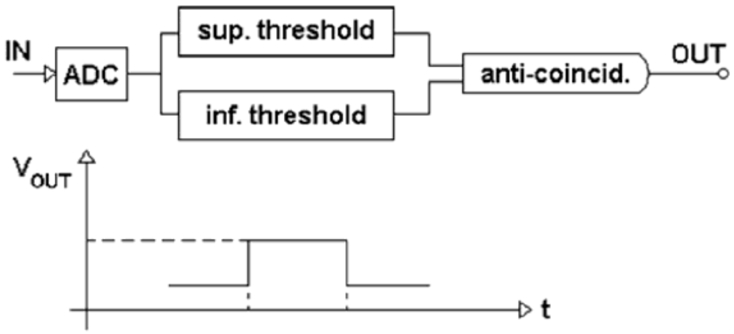


Figure 3. Analogue-to-digital converter, integral discriminators and anti-coincidence circuit.

The good points of a differential discriminator are the rapidity and the stability of the amplitude range; the latter characteristic depends on the thresholds stability and it is function of the same range position [4].

The FET output impulse, which is proportional to the incident X-rays intensity, is still an analogue signal. A *multi-channel analyser* is necessary, then which consists in a certain number of differential discriminators. To assign impulse amplitude to a channel represents an analogue-digital conversion operation. The devices adopted for such purpose are denominated flash ADC. An example of analogue-digital conversion is reported in Figure 4 [14].

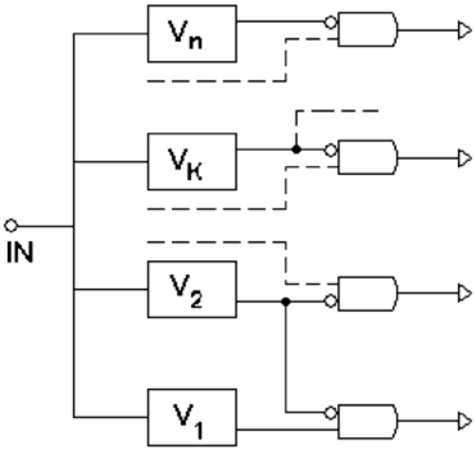


Figure 4. Flash ADC [14].

Such devices are fast, but not enough linear and the limit of their channels number is ~100. The “Wilkinson” type device is the most employed and it reduces the conversion into a time measurement. A capacity is charged, in the same device, than it is linearly discharged. A high frequency oscillator performs the impulses count during the discharge, till the capacity voltage is zero; the number of the counted impulses corresponds to the channels number. The conversion time magnitude order of these ADCs is of the μs, with a 100MHz frequency. The capacity charge and discharge graph is reported in Figure 5.

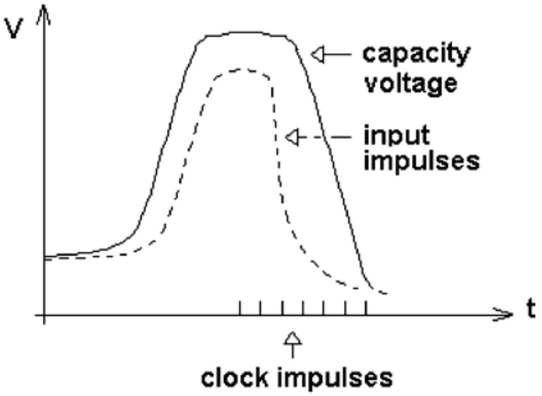


Figure 5. Capacity charge and discharge.

An ADC device frequently employed is the “successive approximations” converter, in which the impulse is compared with a series of “weights”, following an order sequence. The impulse amplitude V is first compared with the weight $E/2$, being E the maximum amplitude. If $V > E/2$, a “1” is created and the difference $V - E/2$ is sent to the 2nd stadium, where it is compared with $E/4$. Then, if $V < E/4$, a “0” is created and the signal passes to the 3rd stadium, where it is compared with $E/8$, etc. The same ADC has a conversion time magnitude order of the μsec, but it has some linearity limits due to the scarce precision of the weights.

The block diagram of the signal processing is well represented in Figures 6 and 7. The basic scheme is reported in Figure 6 [14], where V = analogue voltage to be converted; C = comparator; VR = reference voltage; VF = feedback voltage. Moreover,

$$q = {}^{VR}\!/_{2^n}, \tag{1}$$

where q is the conversion quantum;

$$\varepsilon = |V - VF|, \tag{2}$$

where ε is the error, being always $\varepsilon < q$.

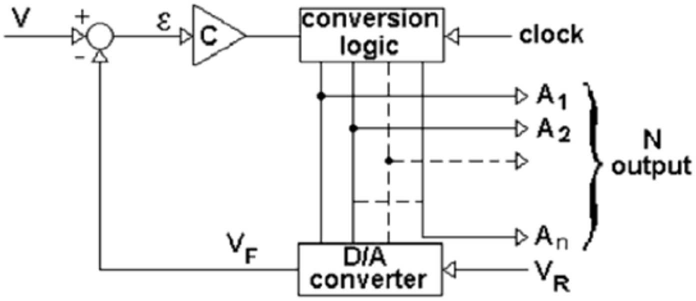


Figure 6. Successive approximations ADC [14].

The “successive approximations” ADC operates according to the “attempt and correction” theory, departing from the most significant bit, which is set equal to 1. The so obtained output $(VF = VR/2)$ is compared to the voltage V of the comparator input. The most significant bit will be reset to 0, if $V < VR/2$. In the contrary case, the same bit will be unchanged. The procedure is extended to the other bits and the process ends when $V \approx VF$, taking into account, the conversion quantum q. The conversion logic can be realized, for instance, through a microprocessor (μP). An example of a 4 bit output (A4, A3, A2 and A1) concerning the conversion diagram into the time dominion is represented in Figure 7 [14].

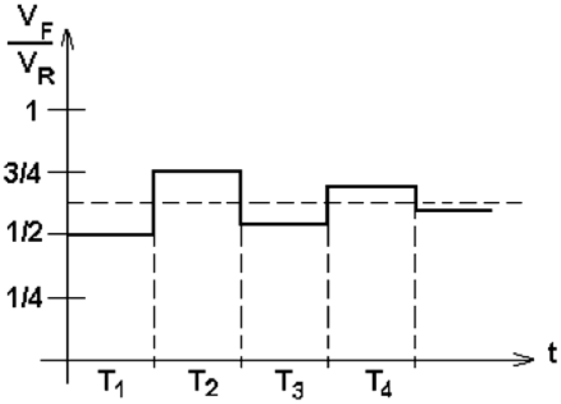


Figure 7. Conversion diagram into the time dominion [14].

2.2. Integral discriminators, anti-coincidence circuit and decade counter

Each integral discriminator, one for the inferior threshold and one for the superior (ref. Figure 3), can be realized as follows. A Schmitt trigger circuit can be employed, which consists in a bi-stable multivibrator (flip-flop) that can reach the commutation by a signal of any form. The same trigger commutes from the high to the low level, when the input signal reaches the $V1$ threshold, and it commutes from the low to the high level, when the input signal reaches the $V2$ threshold, as shown in Figure 8.

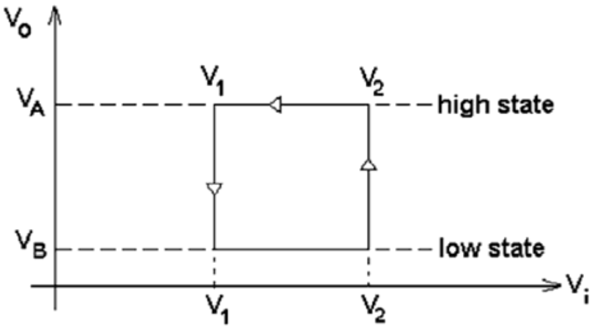


Figure 8. Schmitt trigger commutation scheme.

Figure 9 represents the Schmitt trigger symbol, while Figure 10 shows its functional diagram [1, 12].

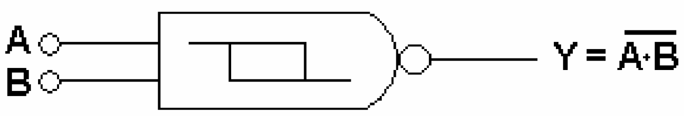


Figure 9. Schmitt trigger symbol.

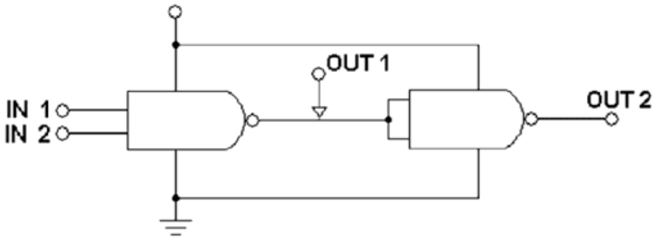


Figure 10. Schmitt trigger functional diagram.

The Schmitt trigger time dominion, input and output signals are represented in Figure 11. Low power Schmitt trigger circuit exists, where the first circuit is a strictly low power, whereas the second and third circuits are derived from the first one and provide smaller hysteresis width [1].

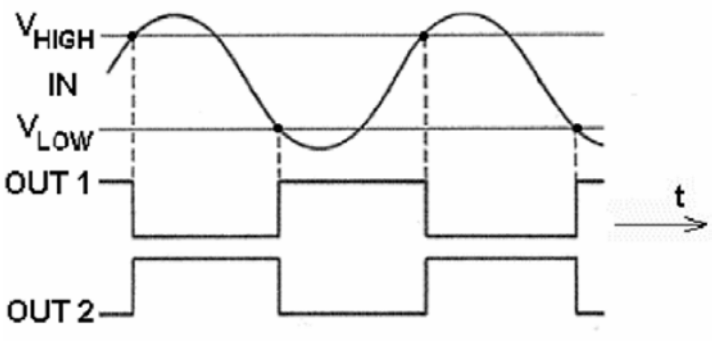
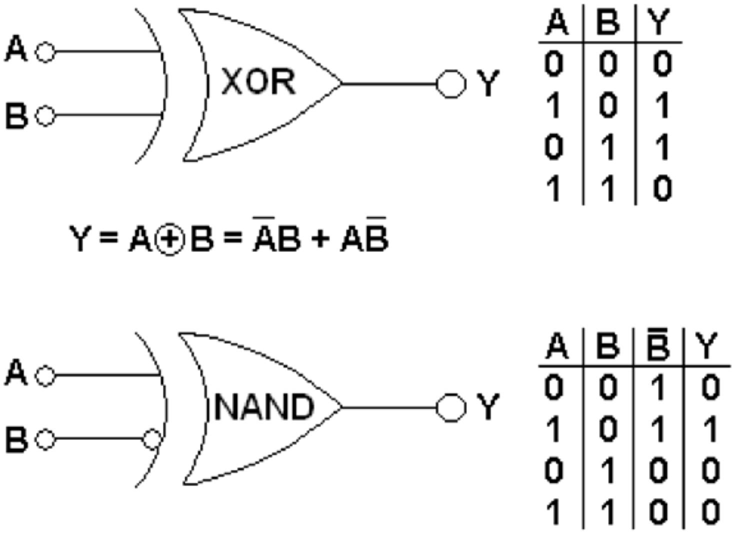


Figure 11. Schmitt trigger time dominion, input and output signals.

The classical anti-coincidence circuit is built by using a XOR-gate, whose output is given by the following logical relation:

$$Y = \overline{A}B + A\overline{B}. \tag{3}$$

The corresponding symbol and table are reported in Figure 12.



A	B	Y
0	0	0
1	0	1
0	1	1
1	1	0

$$Y = A \oplus B = \overline{A}B + A\overline{B}$$

A	B	$\overline{B}$	Y
0	0	1	0
1	0	1	1
0	1	0	0
1	1	0	0

Figure 12. XOR-gate symbol and table.

In this case, however, it is preferable to use an AND-gate with a negative output, because the configuration $A = 1$, $B = 0$, $Y = 1$ must be excluded [11].

The decade counter is a four bit up binary counter. It adopts four flip-flop series connected, which can be of *T* or *D* type with a Q to *D* feedback, or *JK* type $(J = K = 1)$.

A decade counter is thus created, which is able to count from 0 to 9 and then, at the tenth impulse, it is reset through the AND-gate. The considered count, without AND-gate, would reach 15 with the consequent resetting. Such count would start again with the seventeenth pulse, but valued as the first. If the following configuration occurs:

$$Q3 = 1;\ Q2 = 0;\ Q1 = 1;\ Q0 = 0, \tag{4}$$

the $Q3$ and $Q1$ outputs, both to the logical 1, enter in the AND-gate. The AND-gate output will thus reach the logical 1, resetting the outputs of all the flip-flop. Figure 13 reports the functional diagram of the decade counter, while Figure 14 shows the decade counter operation due to a clock signal and to a gating signal both present in a NAND-gate.

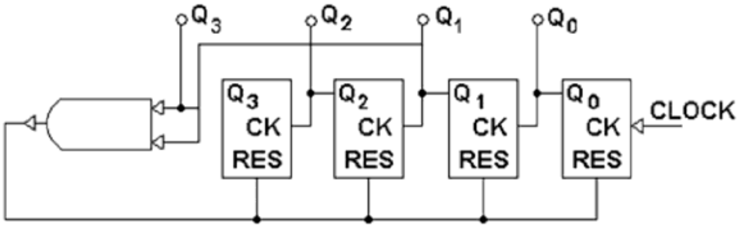


Figure 13. Decade counter functional diagram.

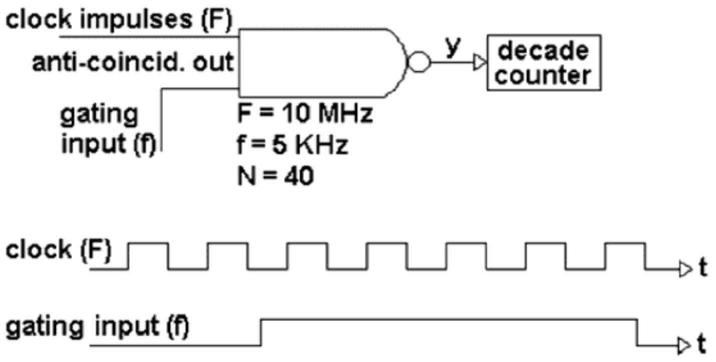


Figure 14. Decade counter operation.

Since from the logical 1 trained NAND-gate inverted pulses go out, a "negative edge triggered" counter must be used [11].

3. Experimental and Discussion

The Si(Li) detector is widely used in the field of elemental analysis. The detection of secondary radiation (also called "*fluorescent*") from a specimen irradiated by X-rays (or gamma rays) offers the double possibility of counting the photons emitted by the atoms of the sample, discriminating the energies of the characteristic lines. The time required for a complete analysis, consequently, can be drastically reduced without a significant lost of accuracy. The continuously improved resolution of Si(Li) detectors, in fact, allows reaching detection limits of the order of ppb and ppt, depending on the geometry of the spectrometer. Figure 15 shows an X-ray fluorescence (XRF) spectrum, we have obtained in geometry of "total reflection" (the incident beam, from the X-ray tube, has an inclination of few seconds of arc with respect to the surface of the sample). The adopted source was a monochromatic $\mathrm{MoK}\alpha$ radiation. The peaks shown in this figure correspond to the excitation of 50ppm of Ga and 50ppm of Mn.

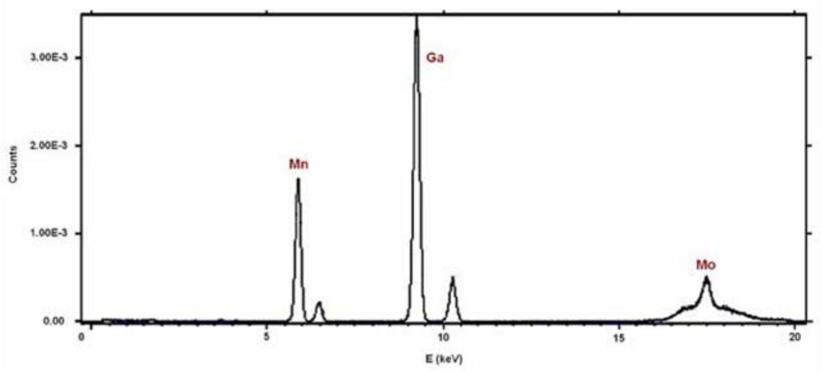


Figure 15. XRF spectrum.

The X-ray detection technique by Si(Li) crystal semiconductor, in comparison with an "ionization chamber", in which the X-rays ionize a gas rather than a semiconductor, requires, for the formation of an electron-hole pair, energy of an inferior order of magnitude, and this represents its main advantage.

For the FET, that gives an output voltage impulse, the main problem is the thermal noise, which decreases to the low temperatures. The same transistor must be maintained therefore to the liquid N temperature and it has to be also protected from the light, to which it results sensitive. The impulse, measured at its output, is proportional to the X-ray photons energy, and it introduces 0.05% stability with reference to the operative voltage.

The considered technique adopts electronic supports easily feasible, and its future developments in industrial sectors are foreseen. The characterisation of approximately isotropic poly-crystalline materials of technological interest includes: Diffractometry methods for the superficial residual stress measurement, characterisation of crystalline structures, identification of the crystalline phases, determination of the crystallites dimensions, determination of the degree of order of the alloys, structure studies of coatings, thin film analysis, and reflectometry.

The qualitative and qualitative elemental analysis is also possible by using the Si(Li) detectors in a XRF fluorescence spectrometer. The possibility of obtaining very good detection limits allows the analysis of contaminants in the electronic industry.

The use of Si(Li) detectors will probably increase, when cooling devices will be available with improved performances, in comparison with traditional ones, such as the ionization chambers and the scintillation counters: The first adopts cylinders filled with gas and provided of a Be window (a voltage is measured proportional to the incidental X-rays energy), the latter a NaI crystal, absorbing X-rays and emitting a bright radiation amplified through a photomultiplier.

A user's overview of the applications of X-ray detectors for material's investigation is given in [2].

The intrinsic efficiency of a Si(Li) detector has been recently simulated with the Monte Carlo codes, showing an appreciable agreement between the simulations and experimental measurements with several point-like sources [8].

An electrical and nuclear characterisation has been recently carried out concerning conventional Si(Li) detectors realized on Topsil silicon. The preliminary results show that, the said detectors present good electrical and nuclear characteristics that can be employed in X-ray spectrometry and extensive applications in research science, environment monitoring, and natural radioactivity. The central contribution of this work is to show an easy-to-implement, low cost detector set, achievable by adopting a not expensive $n^{+}p$ diode [6].

A considerable progress has been achieved in the advance of Si(Li) detectors functioning without liquid nitrogen, with a working temperature maintainable by thermoelectric Peltier's coolers [13].

Conclusions

The Si(Li) detector, in the past, has been adopted for a not relevant number of applications in the diffractometry. In this field, Na(I) scintillation counters have been historically used, successively followed by new devices such as position sensitive (PSD), charge-coupled (CCD), diode-array (DAD), and area detectors.

Si(Li) detectors can be currently considered among the preferable type of sensors for X-ray registration. Their high energy resolution and registration efficiency make them the most helpful for applications in X-ray diffractometry, fluorescence analysis, and other scientific fields.

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